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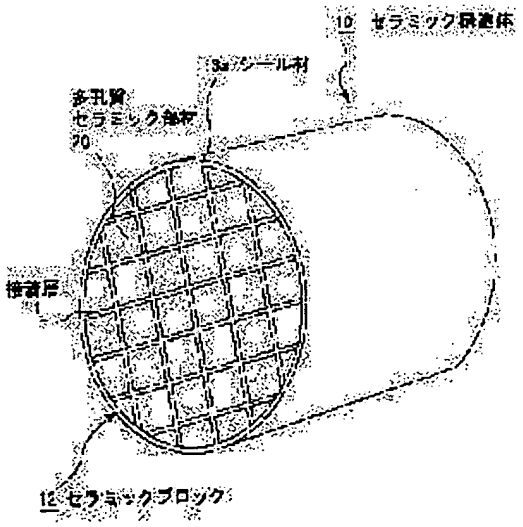
(71)Applicant : IBIDEN CO LTD

(72)Inventor : ONO KAZUSHIGE
YOSHIDA YOSHIYUKI

(54) CERAMICS STRUCTURE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide durable ceramics structure that a piled particulate in a reclaiming process is burned and removed perfectly because of its high thermal conductivity of an adhesive layer binding several ceramics components and no crack by vibration and pressure of an exhaust gas occurs in the adhesive layer because of its high adhesive strength.



SOLUTION: The ceramics block is composed by binding several square rod-shaped components of the porous ceramics where longitudinally continuous pores separated with partition are arranged

to the adhesive layers. The partitions work as a particle-collecting filter and the adhesive layers contain at least an inorganic binder, an organic binder and a silicon carbide fiber.

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(71) **Applicant**
Identification Number 000000158
Name IBIDEN CO., LTD.
Address 2-1, Kanda-cho, Ogaki-shi, Gifu-ken
(72) **Inventor(s)**
Name Ono Kazushige
Address 1-1, Kitagata, Ibigawa-cho, Ibi-gun, Gifu-ken Inside of IBIDEN Ogaki North Works
(72) **Inventor(s)**
Name Yoshida Right line
Address 1-1, Kitagata, Ibigawa-cho, Ibi-gun, Gifu-ken Inside of IBIDEN Ogaki North Works
(74) **Attorney**
Identification Number 100086586
Patent Attorney
Name Yasutomi Yasuo (outside binary name)
Theme code (reference)

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(57) **Abstract**
Technical problem Since the heat conductivity of the glue line which bands two or more ceramic

members together is high, while being able to carry out combustion clearance of the particulate deposited in the regeneration thoroughly, since the bond strength is also large, the ceramic structure which is excellent in the endurance which a crack etc. produces in a glue line neither with an oscillation nor the pressure of exhaust gas is offered.
Means for Solution It is the ceramic structure characterized by being the ceramic structure which two or more porosity ceramic members of the prism configuration by which many breakthroughs separated the septum and were installed in the longitudinal direction side by side banded together through the glue line, and constituted the hollow clay building block, and was constituted so that the septum which separates the above-mentioned breakthrough might function as a filter for particle uptake, and the above-mentioned glue line containing an inorganic binder, an organic binder, and silicon carbide fiber at least.

Claim(s)
Claim 1 It is the ceramic structure characterized by being the ceramic structure which two or more porosity ceramic members of the prism configuration by which many breakthroughs separated the septum and were installed in the longitudinal direction side by side banded together through the glue line, and constituted the hollow clay building block, and was constituted so that the septum which separates said breakthrough might function as a filter for particle uptake, and said glue line containing an inorganic binder, an organic binder, and silicon carbide fiber at least.

Detailed Description of the Invention
0001
Field of the Invention This invention relates to the ceramic structure used as a filter from which the particulate in the exhaust gas discharged by the internal combustion engine etc. is removed.
0002
Description of the Prior Art It poses a problem that the particulate contained in the exhaust gas discharged by internal combustion engines, such as cars, such as a bus and a truck, and a construction equipment, does damage to an environment or the body recently. By passing a porosity ceramic for this exhaust gas, the ceramic filter which carries out uptake of the particulate in exhaust gas, and purifies exhaust gas is proposed variously.
0003 As for the ceramic structure which constitutes these ceramic filters, many breakthroughs are usually installed in an one direction side by side, and the septum which separates breakthroughs functions as a filter. That is, after the exhaust gas with which, as for the breakthrough formed in the ceramic structure, either the entry side of exhaust gas or the edge of an outlet side flowed into ********** and the breakthrough of 1 with the filler passes the septum which surely separates a breakthrough, in case it flows out of other breakthroughs and exhaust gas passes this septum, a particulate is caught in a septum part and exhaust gas is purified.
0004 In connection with the cleaning effect of such exhaust gas, a particulate accumulates on the septum part which separates the breakthrough of the ceramic structure gradually, blinding is started into it, and it comes to bar aeration into it. For this reason, this ceramic filter needs to carry out combustion clearance of the particulate which causes blinding using heating means, such as a heater, periodically, and needs to be reproduced.
0005 However, in this regeneration, uniform heating of the ceramic structure is difficult, and since local generation of heat accompanying particulate combustion occurs, big thermal stress occurs. Moreover, by the thermal shock which the rapid temperature change of exhaust gas gives at the time of the usual operation, uneven temperature distribution arise inside the ceramic structure, and thermal stress occurs. Consequently, when the above-mentioned ceramic structure consisted of single ceramic members, the crack occurred and there was a trouble of giving the serious trouble for particulate uptake.
0006 Therefore, for example, the particulate trap which reduced the thermal stress which acts on the ceramic structure is indicated by JP,60-65219,A by dividing the ceramic structure into two or more ceramic members.

0007 Moreover, when banding two or more ceramic members, the sealant of a non-adhesive property is made to insert in the clearance produced between each part material, and the particle uptake filter which prevented that exhaust gas leaked from the clearance between the ceramic structures is indicated by the publication of unexamined utility model application Heisei 1-63715 official report.
0008 However, although generating and destruction of a crack resulting from thermal stress could be prevented with the particle uptake filter indicated by this publication of unexamined utility model application Heisei 1-63715 official report, there was a trouble that each ceramic member was firmly unjoinable.
0009 Moreover, in the regeneration which generally carries out combustion clearance of the deposited particulate, the temperature near the center of the ceramic structure tends to become high compared with the temperature near **the** the rim section. However, in the conventional ceramic structure, since the heat conductivity between the ceramic members which constitute this ceramic structure was not not much high, the particulate deposited near **the** the rim section also had the cinder and the problem that it was difficult to remove thoroughly.
0010 In order to solve such a problem, this invention persons developed the ceramic structure joined by the sealant (glue line) in which each ceramic member which is indicated by JP,8-28246,A contains an inorganic fiber, an inorganic binder, a heat-resistant organic binder, a heat-resistant inorganic particle, etc. previously.
0011 According to the effectiveness of a tangle to the inorganic fiber in the sealant (glue line) which joins each ceramic members, an organic binder, and an inorganic fiber and an inorganic binder, the ceramic structure which such this invention persons developed previously was also able to secure the thermal conductivity of a sealant (glue line) while bond strength had been improved to some extent.
0012 However, since the above-mentioned inorganic particle was not able to check a tangle to an inorganic fiber and an inorganic binder and was not able to keep the bond strength high enough, a crack etc. may arise in a glue line with an oscillation, the pressure of exhaust gas, etc., and there was a fixed limitation in both securing the bond strength and the heat conductivity of the sealant (glue line) on high level in such the ceramic structure.
0013
Problem(s) to be Solved by the Invention This invention was made in order to solve these problems, and since the bond strength is also large, it aims at offering the ceramic structure which is excellent in the endurance which a crack etc. produces in a glue line neither with an oscillation nor the pressure of exhaust gas, while it can carry out combustion clearance of the deposited particulate thoroughly in the regeneration, since its heat conductivity of the glue line which bands two or more ceramic members together is high.
0014
Means for Solving the Problem The ceramic structure of this invention is the ceramic structure which two or more porosity ceramic members of the prism configuration by which many breakthroughs separated the septum and were installed in the longitudinal direction side by side banded together through the glue line, and constituted the hollow clay building block, and was constituted so that the septum which separates the above-mentioned breakthrough might function as a filter for particle uptake, and the above-mentioned glue line is characterized by including an inorganic binder, an organic binder, and silicon carbide fiber at least.
0015
Embodiment of the Invention Hereafter, the operation gestalt of the ceramic structure of this invention is explained based on a drawing.
0016 Two or more porosity ceramic members of the prism configuration by which many breakthroughs separated the septum and were installed in the longitudinal direction side by side band together through a glue line, and constitute a hollow clay building block, the ceramic structure of this invention is constituted so that the septum which separates the above-mentioned breakthrough may function as a filter for particle uptake, and this glue line contains an inorganic binder, an organic binder, and silicon carbide fiber at least.
0017 Drawing 1 is the perspective view having shown typically 1 operation gestalt of the ceramic structure of this invention, and drawing 2 is the perspective view having shown typically the porosity ceramic member which constitutes the ceramic structure of this invention.

0018 As shown in drawing 2 , many breakthroughs 21 are formed in the porosity ceramic member 20 which constitutes the ceramic structure, and, as for the end section of the porosity ceramic member 20 which has these breakthroughs 21, the checker is filled up with the filler 22. Moreover, in other edges which are not illustrated, the breakthrough 21 with which the filler is not filled up into the end section is filled up with the filler.
0019 Drawing 1 shows the ceramic structure 10 which banded two or more porosity ceramic members 20 shown in drawing 2 . Moreover, in drawing 1 , the breakthrough 21 formed in the porosity ceramic member 20 is omitted.
0020 In this ceramic structure 10, two or more porosity ceramic members 20 band together through a glue line 11, and constitute a hollow clay building block 12, and this glue line 11 contains an inorganic binder, an organic binder, and silicon carbide fiber at least. Moreover, coating of the sealant 13a is carried out to the whole periphery section of a hollow clay building block 12, and the ceramic structure 10 is formed. Although especially the configuration of the above-mentioned ceramic structure is not limited but the shape of a cylindrical shape and a prism configuration are also available, as shown in drawing 1 , the cylindrical shape-like thing is usually used well.
0021 Since only any 1 edge is filled up with the filler 22 as the breakthrough 21 of a large number which constitute this ceramic structure 10 was shown in drawing 2 , the exhaust gas which flowed from the end section of the breakthrough 21 of 1 which is carrying out opening surely passes the septum 23 of the porosity which separates between the adjoining breakthroughs 21, and flows out through other breakthroughs 21. And in case exhaust gas passes a septum 23, the particulate in exhaust gas will be caught.
0022 Although especially the construction material of the porosity ceramic member which constitutes the above-mentioned ceramic structure 10 is not limited but various ceramics are mentioned, in these, thermal resistance is large, it excels in a mechanical property and large silicon carbide of thermal conductivity is desirable.
0023 Although especially the particle size of these ceramics is not limited, either, what combined the powder 100 weight section which has the mean particle diameter which what has few contraction is desirable, for example, is about 0.3-50 micrometers, and the powder 5 - 65 weight sections which have the mean particle diameter of about 0.1-1.0 micrometers at the next baking process is desirable. Moreover, although especially the ingredient that constitutes sealant 13a is not limited, either, the thing containing heat-resistant ingredients, such as an inorganic fiber and an inorganic binder, is desirable. Sealant 13a may be constituted by the same ingredient as a glue line 11.
0024 The ingredient which constitutes a glue line 11 contains an inorganic binder, an organic binder, and silicon carbide fiber. Although the bond strength of a glue line 11 is improved according to the effectiveness of a tangle to the silicon carbide fiber in a glue line 11, an inorganic binder, and silicon carbide fiber and an organic binder, both the bond strength of a glue line 11 and the heat conductivity are securable on high level by making silicon carbide fiber contain. It is thought by the reason being that the touch area of these silicon carbide fiber in the inside of a glue line increases since the silicon carbide to add is fibrous, and its heat conductivity improving compared with the silicon carbide particle added conventionally, and not checking effectiveness of a tangle to an inorganic binder etc. and becoming entangled with both reverse, since it is fibrous that the bond strength of a glue line 11 becomes high.
0025 As the above-mentioned inorganic binder, a silica sol, alumina sol, etc. are mentioned, for example. These may be used independently and may use two or more sorts together. In these, a silica sol is desirable.
0026 As the above-mentioned organic binder, polyvinyl alcohol, methyl cellulose, ethyl cellulose, a carboxy cellulose, etc. are mentioned, for example. These may be used independently and may use two or more sorts together. In these, a carboxy cellulose is desirable.
0027 As for the fiber length of the above-mentioned silicon carbide fiber, it is desirable that it is 20-300 micrometers, and it is more desirable that it is 50-200 micrometers. The property becomes it close to a particle that fiber length is less than 20 micrometers, and lowering of bond strength is caused. On the other hand, if it exceeds 300 micrometers, into a glue line, it will become difficult to distribute homogeneity and it will cause lowering of bond strength too. Moreover, as for the diameter of fiber, it is desirable that it is 3-15 micrometers. Since the reinforcement of silicon carbide fiber will fall that the diameter of fiber is less than 3 micrometers and it will be cut easily,

lowering of bond strength is caused. On the other hand, if it exceeds 15 micrometers, it will be difficult to check a tangle to an inorganic binder, and to cause lowering of bond strength, and to obtain such thick silicon carbide fiber itself, and it will cause the jump of raw material cost.

0028 It is solid content, the content of the inorganic binder in a glue line 11 has 1 - 40 desirable % of the weight, its 1 - 20 % of the weight is more desirable, and its 5 - 15 % of the weight is still more desirable. Lowering of bond strength is caused as the content of an inorganic binder is less than 1 % of the weight, and on the other hand, if it exceeds 40 % of the weight, decline in thermal conductivity will be caused.

0029 It is solid content, the content of the organic binder in a glue line 11 has 0.1 - 5.0 desirable % of the weight, its 0.2 - 1.0 % of the weight is more desirable, and its 0.4 - 0.8 % of the weight is still more desirable. It becomes difficult to control the migration of a glue line 11 as the content of an organic binder is less than 0.1 % of the weight, on the other hand, when it exceeded 5.0 % of the weight and a glue line 11 is exposed to an elevated temperature, an organic binder is burned down and bond strength falls.

0030 It is solid content, the content of the silicon carbide fiber in a glue line 11 has 3 - 80 desirable % of the weight, its 10 - 70 % of the weight is more desirable, and its 40 - 60 % of the weight is still more desirable. Decline in thermal conductivity is caused as the content of silicon carbide fiber is less than 3 % of the weight, and on the other hand, when it exceeded 80 % of the weight and a glue line 11 is exposed to an elevated temperature, lowering of bond strength is caused.

0031 Although little moisture, a little solvent, etc. may be included besides an inorganic binder, an organic binder, and silicon carbide fiber in the glue line 11, such moisture, a solvent, etc. usually almost disperse with heating after applying a glue line paste etc.

0032 Since it is what contains an inorganic binder, an organic binder, and silicon carbide fiber in the glue line to which the ceramic structure of this invention bands two or more ceramic members together as above-mentioned, it becomes the thing excellent in both the thermal conductivity and bond strength. Therefore, in the regeneration, the ceramic structure of this invention becomes what a crack arose in a glue line neither with an oscillation nor the pressure of exhaust gas, and was excellent in endurance while being able to carry out combustion clearance of the deposited particulate thoroughly.

0033 Next, the manufacture approach of the ceramic structure of this invention is explained. In addition, suppose that silicon carbide is used as a raw material of the ceramic member which constitutes the ceramic structure in the following explanation.

0034 A silicon carbide Plastic solid is produced first. After mixing silicon carbide powder, a binder, and dispersion-medium liquid and preparing the mixed constituent for Plastic solid manufacture in this process, by performing extrusion molding of this mixed constituent The column-like silicon carbide Plastic solid with which many breakthroughs separated the septum and were installed in the longitudinal direction side by side is produced, by drying this Plastic solid after this, dispersion-medium liquid is evaporated and the silicon carbide Plastic solid containing silicon carbide powder and resin is produced. In addition, little dispersion-medium liquid may be contained in this silicon carbide Plastic solid.

0035 The configuration of the appearance of this silicon carbide Plastic solid is isomorphism-like mostly with the porosity ceramic member 20 shown in drawing 2 , and also may have the shape of the shape of an elliptic cylinder, or the triangle pole etc. In addition, at this process, the part equivalent to a filler 22 serves as a cavity.

0036 It is not limited especially as the above-mentioned binder, for example, methyl cellulose, a carboxymethyl cellulose, hydroxyethyl cellulose, a polyethylene glycol, phenol resin, an epoxy resin, etc. can be mentioned. The loadings of the above-mentioned binder usually have desirable 1 - 10 weight section extent to the above-mentioned silicon carbide powder 100 weight section.

0037 It is not limited especially as the above-mentioned dispersion-medium liquid, for example, alcohol **, such as an organic solvent; methanol,** , such as benzene, water, etc. can be mentioned. Optimum dose combination of the above-mentioned dispersion-medium liquid is carried out so that the viscosity of the above-mentioned resin may become fixed within the limits.

0038 Next, the process which obturates the above-mentioned breakthrough of the produced silicon carbide Plastic solid in the shape of an obturation pattern with a restoration paste as an obturation process is performed. In this case, some breakthroughs are obturated with a restoration

paste by contacting the breakthrough of a silicon carbide Plastic solid in the mask with which puncturing was formed in the shape of an obturation pattern, and making a restoration paste invade into it from puncturing of the above-mentioned mask at the above-mentioned breakthrough.

0039 Or it will not be the mixed constituent and this appearance which were used as the above-mentioned restoration paste on the occasion of manufacture of a ceramic Plastic solid, what added the dispersion medium further to the above-mentioned mixed constituent is desirable.

0040 Next, the process which pyrolyzes the resin in the silicon carbide Plastic solid produced by the above-mentioned process as a cleaning process is performed. At this cleaning process, after laying the above-mentioned silicon carbide Plastic solid on the fixture for cleaning, it carries in to a cleaning furnace and usually heats at 400-650 degrees C under an oxygen content ambient atmosphere. Thereby, while resinous principles, such as a binder, vaporize, it decomposes and disappears and only silicon carbide powder remains mostly.

0041 Next, the process which lays the degreased silicon carbide Plastic solid on the fixture for baking, and calcinates it as a baking process is performed. At this baking process, the column-like silicon carbide sintered compact with which many breakthroughs separated the septum and were installed in the longitudinal direction side by side is manufactured by heating the silicon carbide Plastic solid degreased at 2000-2200 degrees C under inert gas ambient atmospheres, such as nitrogen and an argon, and making silicon carbide powder sinter.

0042 In addition, at a series of processes of resulting **from a cleaning process** in a baking process, the above-mentioned silicon carbide Plastic solid is carried on the fixture for baking, and it is desirable to perform a cleaning process and a baking process as it is. It is because it can prevent that can perform a cleaning process and a baking process efficiently, and carry, and a silicon carbide Plastic solid gets damaged in a substitute etc.

0043 Thus, many breakthroughs separate a septum and it is installed in a longitudinal direction side by side, and after manufacturing the porosity silicon carbide sintered compact constituted so that the above-mentioned septum might function as a filter, the glue line mentioned above into the outer wall part of a porosity silicon carbide sintered compact is formed as a union process of this porosity silicon carbide sintered compact, more than one band the above-mentioned porosity silicon carbide sintered compact together, and a hollow clay building block is produced so that it may become predetermined magnitude.

0044 Then, manufacture of the ceramic structure of this invention is ended by heating and stiffening **dry and** this hollow clay building block on 50-100 degrees C and the conditions of 1 hour, and forming sealant 13a in that periphery section, after cutting using after that, for example, a diamond cutter etc., almost like the ceramic structure 10 which showed that periphery section to drawing 1 .

0045 By carrying out each process explained above, thermal conductivity is high and the ceramic structure excellent also in the bond strength of each ceramic member can be manufactured.

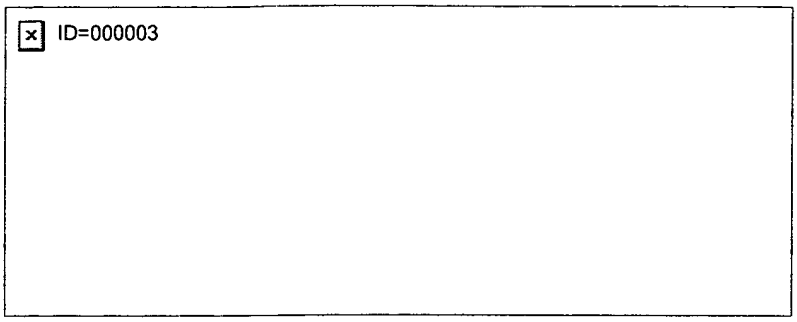
0046

Example Although an example is hung up over below and this invention is explained to it in more detail, this invention is not limited only to these examples.

0047 The mixed constituent of a raw material was prepared by blending the alpha mold silicon carbide powder 70 weight section with example 1 mean particle diameter of 10 micrometers, the beta mold silicon carbide powder 30 weight section with a mean particle diameter of 0.7 micrometers, the methyl cellulose 5 weight section, the dispersant 4 weight section, and the water 20 weight section, and mixing to homogeneity. The extruding press machine was filled up with this mixed constituent, and the generation form of a honeycomb configuration was produced in a part for extrusion rate/of 2cm. This generation form is the same as that of the porosity ceramic member 20 shown in drawing 2 almost, that magnitude is 33mmx33mmx300mm, and, for an average pore diameter, the number of 1-40 micrometers and breakthroughs is 31-/cm2. The thickness of a septum was 0.35mm.

0048 After using the above-mentioned mixed constituent and the bulking agent paste of this component for the desiccation object of this generation form and filling it up with a bulking agent in the predetermined part of the breakthrough of a silicon carbide sintered compact, the porosity silicon carbide member was manufactured by degreasing at 450 degrees C and carrying out heating baking at 2200 degrees C further.

0049 Next, the carboxymethyl cellulose of 0.65 % of the weight, the fiber length of 100-200 micrometers, 44.2 % of the weight of silicon carbide fiber of 3-15 micrometers of diameters of fiber, and 40.15 % of the weight of water were mixed and kneaded as an inorganic binder as 15 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, and an organic binder, and the paste for glue lines was prepared.
0050 Next, the above-mentioned paste for glue lines was stuck on the peripheral face of 1 of the produced porosity silicon carbide member, and the glue line was formed. And after laying other porosity silicon carbide members on this glue line, it was made to dry and harden in 100 degrees C and 1 hour, and the combination of the porosity silicon carbide member which two porosity silicon carbide members combined was produced.
0051 The carboxymethyl cellulose of 0.6 % of the weight, the fiber length of 100-200 micrometers, 60 % of the weight of silicon carbide fiber of 3-15 micrometers of diameters of fiber, and 30.4 % of the weight of water were used as an example 2 inorganic binder as 9 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, and an organic binder, and also the combination of a porosity silicon carbide member was produced like the example 1.
0052 The carboxymethyl cellulose of 0.6 % of the weight, the fiber length of 100-200 micrometers, 44.2 % of the weight of silicon carbide fiber of 3-15 micrometers of diameters of fiber, and 46.2 % of the weight of water were used as an example 3 inorganic binder as 9 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, and an organic binder, and also the combination of a porosity silicon carbide member was produced like the example 1.
0053 13.3 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols and 42.5 % of the weight of water were used as 44.2 % of the weight (shot content: 2.7%, fiber length:30-100mm) of ceramic fiber which consists of an alumina silica as example of comparison 1 inorganic fiber, and an inorganic binder, and also the combination of a porosity silicon carbide member was produced like the example 1. In this example 1 of a comparison, when drying the glue line, the migration of the binder which constitutes a glue line, or a member occurred, and the glue line became an ununiformity and has carried out.
0054 Ceramic fiber which consists of alumina silicate as example of comparison 2 inorganic fiber (shot content: 3%) As an inorganic binder, 23.3 0.1-100mm% of the weight Fiber length : 7 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, 0.5 % of the weight of carboxymethyl celluloses, 30.2 % of the weight of silicon carbide powder with a mean particle diameter of 0.3 micrometers, and 39 % of the weight of water were used as an organic binder, and also the combination of a porosity silicon carbide member was produced like the example 1.
0055 It measured by the approach of showing below the performance evaluation of the combination of the porosity silicon carbide member manufactured in examples 1-3 and the examples 1-2 of a comparison.
0056 As shown in measurement drawing 3 of the assessment approach (1) bond strength, two triangle pole-like members have been arranged on a base, then the above-mentioned combination was laid so that the porosity silicon carbide member of ends might appear on the above-mentioned triangle pole-like member, the load was applied to the main glue line part, and the load when peeling arises in a glue line was measured. Moreover, in the actual activity, since the rapid heating to about room temperature -900 degree C and quenching were expected, assessment with the same said of the thing after performing the thermo-cycle trial (100 times) which is room temperature -900 degree C was performed. The result is shown in the following table 1.
0057 (2) After laying the above-mentioned combination so that two porosity silicon carbide members may be accumulated as shown in measurement drawing 4 of thermal conductivity, the temperature gradient of the upside temperature T1 and the lower temperature T2 was measured by installing the periphery on an enclosure and a heater 31 with a heat insulator 30, and heating for 30 minutes at 600 degrees C. The result is shown in a table 1.
0058
A table 1



0059 Although the typical bond strength of the glue line of the combination of the porosity silicon carbide member concerning examples 1-3 was 2.80-3.30MPa and the temperature gradient of the upper bed and soffit was 23-31 degrees C so that clearly from the result shown in a table 1, it was inferior to the combination of the porosity silicon carbide member which the typical bond strength of the glue line of the combination of the porosity silicon carbide member concerning the examples 1-2 of a comparison requires for 2.14-2.42MPa with 50-80 degrees C, and the temperature gradient all requires for an example. In addition, in this example and the example of a comparison, although what combined only two porosity silicon carbide members was used and the bond strength and thermal conductivity were measured, in order to combine many porosity silicon carbide members, the difference of the value of bond strength and thermal conductivity will become still more remarkable at the actual ceramic structure.

0060

Effect of the Invention It becomes what a crack arose in a glue line neither with an oscillation nor the pressure of exhaust gas, and was excellent in endurance while it can carry out combustion clearance of the deposited particulate thoroughly in the regeneration, since the ceramic structure of this invention is as above-mentioned.

Brief Description of the Drawings

Drawing 1 It is the perspective view having shown typically 1 operation gestalt of the ceramic structure of this invention.

Drawing 2 It is the perspective view having shown typically the porosity ceramic member which constitutes the ceramic structure of this invention.

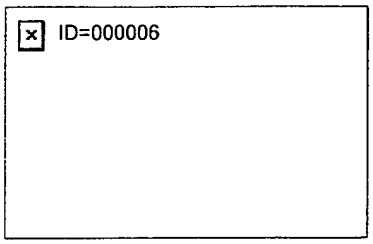
Drawing 3 It is the explanatory view of a measurement trial of bond strength.

Drawing 4 It is the explanatory view of a measurement trial of thermal conductivity.

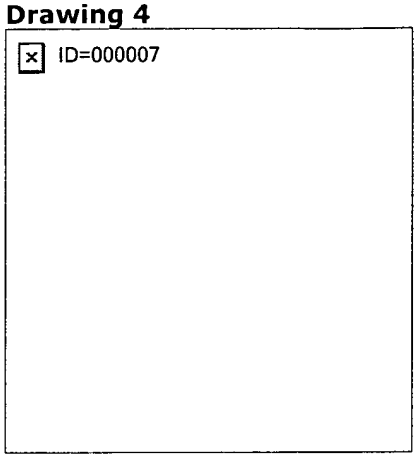
Description of Notations

10 Ceramic Structure
11 Glue Line
12 Hollow Clay Building Block
13 Sealant Paste
13a Sealant
20 Porosity Ceramic Member
21 Breakthrough
22 Filler
23 Septum
30 Heat Insulator
31 Heater

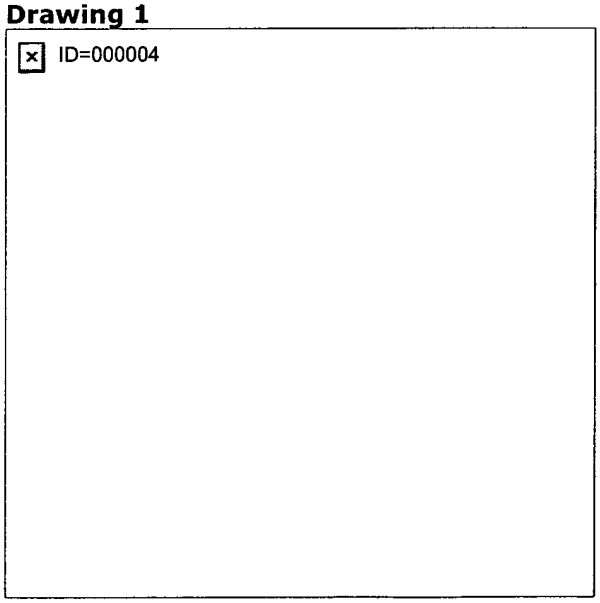
Drawing 3



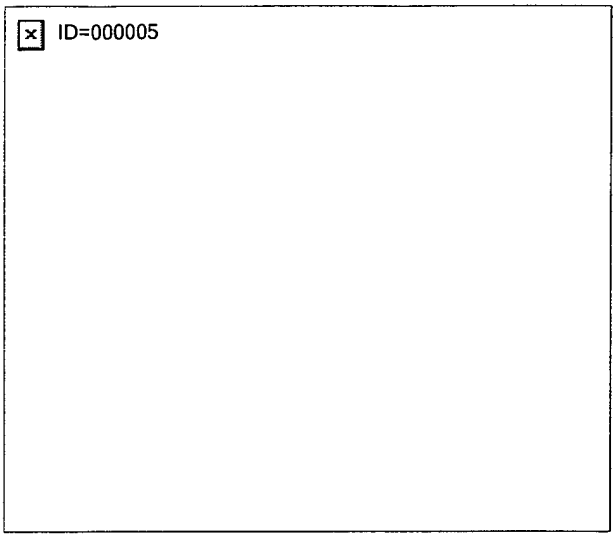
Drawing 4



Drawing 1



Drawing 2



DETAILED DESCRIPTION

Detailed Description of the Invention

0001

Field of the Invention This invention relates to the ceramic structure used as a filter from which the particulate in the exhaust gas discharged by the internal combustion engine etc. is removed.

0002

Description of the Prior Art It poses a problem that the particulate contained in the exhaust gas discharged by internal combustion engines, such as cars, such as a bus and a truck, and a construction equipment, does damage to an environment or the body recently. By passing a porosity ceramic for this exhaust gas, the ceramic filter which carries out uptake of the particulate in exhaust gas, and purifies exhaust gas is proposed variously.

0003 As for the ceramic structure which constitutes these ceramic filters, many breakthroughs are usually installed in an one direction side by side, and the septum which separates breakthroughs functions as a filter. That is, after the exhaust gas with which, as for the breakthrough formed in the ceramic structure, either the entry side of exhaust gas or the edge of an outlet side flowed into ********** and the breakthrough of 1 with the filler passes the septum which surely separates a breakthrough, in case it flows out of other breakthroughs and exhaust gas passes this septum, a particulate is caught in a septum part and exhaust gas is purified.

0004 In connection with the cleaning effect of such exhaust gas, a particulate accumulates on the septum part which separates the breakthrough of the ceramic structure gradually, blinding is started into it, and it comes to bar aeration into it. For this reason, this ceramic filter needs to carry out combustion clearance of the particulate which causes blinding using heating means, such as a heater, periodically, and needs to be reproduced.

0005 However, in this regeneration, uniform heating of the ceramic structure is difficult, and since local generation of heat accompanying particulate combustion occurs, big thermal stress occurs. Moreover, by the thermal shock which the rapid temperature change of exhaust gas gives at the time of the usual operation, uneven temperature distribution arise inside the ceramic structure, and thermal stress occurs. Consequently, when the above-mentioned ceramic structure consisted of single ceramic members, the crack occurred and there was a trouble of giving the serious trouble for particulate uptake.

0006 Therefore, for example, the particulate trap which reduced the thermal stress which acts on the ceramic structure is indicated by JP,60-65219,A by dividing the ceramic structure into two or more ceramic members.

0007 Moreover, when banding two or more ceramic members, the sealant of a non-adhesive property is made to insert in the clearance produced between each part material, and the particle

uptake filter which prevented that exhaust gas leaked from the clearance between the ceramic structures is indicated by the publication of unexamined utility model application Heisei 1-63715 official report.

0008 However, although generating and destruction of a crack resulting from thermal stress could be prevented with the particle uptake filter indicated by this publication of unexamined utility model application Heisei 1-63715 official report, there was a trouble that each ceramic member was firmly unjoinable.

0009 Moreover, in the regeneration which generally carries out combustion clearance of the deposited particulate, the temperature near the center of the ceramic structure tends to become high compared with the temperature near **the** the rim section. However, in the conventional ceramic structure, since the heat conductivity between the ceramic members which constitute this ceramic structure was not not much high, the particulate deposited near **the** the rim section also had the cinder and the problem that it was difficult to remove thoroughly.

0010 In order to solve such a problem, this invention persons developed the ceramic structure joined by the sealant (glue line) in which each ceramic member which is indicated by JP,8-28246,A contains an inorganic fiber, an inorganic binder, a heat-resistant organic binder, a heat-resistant inorganic particle, etc. previously.

0011 According to the effectiveness of a tangle to the inorganic fiber in the sealant (glue line) which joins each ceramic members, an organic binder, and an inorganic fiber and an inorganic binder, the ceramic structure which such this invention persons developed previously was also able to secure the thermal conductivity of a sealant (glue line) while bond strength had been improved to some extent.

0012 However, since the above-mentioned inorganic particle was not able to check a tangle to an inorganic fiber and an inorganic binder and was not able to keep the bond strength high enough, a crack etc. may arise in a glue line with an oscillation, the pressure of exhaust gas, etc., and there was a fixed limitation in both securing the bond strength and the heat conductivity of the sealant (glue line) on high level in such the ceramic structure.

0013

Problem(s) to be Solved by the Invention This invention was made in order to solve these problems, and since the bond strength is also large, it aims at offering the ceramic structure which is excellent in the endurance which a crack etc. produces in a glue line neither with an oscillation nor the pressure of exhaust gas, while it can carry out combustion clearance of the deposited particulate thoroughly in the regeneration, since its heat conductivity of the glue line which bands two or more ceramic members together is high.

0014

Means for Solving the Problem The ceramic structure of this invention is the ceramic structure which two or more porosity ceramic members of the prism configuration by which many breakthroughs separated the septum and were installed in the longitudinal direction side by side banded together through the glue line, and constituted the hollow clay building block, and was constituted so that the septum which separates the above-mentioned breakthrough might function as a filter for particle uptake, and the above-mentioned glue line is characterized by including an inorganic binder, an organic binder, and silicon carbide fiber at least.

0015

Embodiment of the Invention Hereafter, the operation gestalt of the ceramic structure of this invention is explained based on a drawing.

0016 Two or more porosity ceramic members of the prism configuration by which many breakthroughs separated the septum and were installed in the longitudinal direction side by side band together through a glue line, and constitute a hollow clay building block, the ceramic structure of this invention is constituted so that the septum which separates the above-mentioned breakthrough may function as a filter for particle uptake, and this glue line contains an inorganic binder, an organic binder, and silicon carbide fiber at least.

0017 Drawing 1 is the perspective view having shown typically 1 operation gestalt of the ceramic structure of this invention, and drawing 2 is the perspective view having shown typically the porosity ceramic member which constitutes the ceramic structure of this invention.

0018 As shown in drawing 2 , many breakthroughs 21 are formed in the porosity ceramic member 20 which constitutes the ceramic structure, and, as for the end section of the porosity ceramic

member 20 which has these breakthroughs 21, the checker is filled up with the filler 22. Moreover, in other edges which are not illustrated, the breakthrough 21 with which the filler is not filled up into the end section is filled up with the filler.

0019 Drawing 1 shows the ceramic structure 10 which banded two or more porosity ceramic members 20 shown in drawing 2 . Moreover, in drawing 1 , the breakthrough 21 formed in the porosity ceramic member 20 is omitted.

0020 In this ceramic structure 10, two or more porosity ceramic members 20 band together through a glue line 11, and constitute a hollow clay building block 12, and this glue line 11 contains an inorganic binder, an organic binder, and silicon carbide fiber at least. Moreover, coating of the sealant 13a is carried out to the whole periphery section of a hollow clay building block 12, and the ceramic structure 10 is formed. Although especially the configuration of the above-mentioned ceramic structure is not limited but the shape of a cylindrical shape and a prism configuration are also available, as shown in drawing 1 , the cylindrical shape-like thing is usually used well.

0021 Since only any 1 edge is filled up with the filler 22 as the breakthrough 21 of a large number which constitute this ceramic structure 10 was shown in drawing 2 , the exhaust gas which flowed from the end section of the breakthrough 21 of 1 which is carrying out opening surely passes the septum 23 of the porosity which separates between the adjoining breakthroughs 21, and flows out through other breakthroughs 21. And in case exhaust gas passes a septum 23, the particulate in exhaust gas will be caught.

0022 Although especially the construction material of the porosity ceramic member which constitutes the above-mentioned ceramic structure 10 is not limited but various ceramics are mentioned, in these, thermal resistance is large, it excels in a mechanical property and large silicon carbide of thermal conductivity is desirable.

0023 Although especially the particle size of these ceramics is not limited, either, what combined the powder 100 weight section which has the mean particle diameter which what has few contraction is desirable, for example, is about 0.3-50 micrometers, and the powder 5 - 65 weight sections which have the mean particle diameter of about 0.1-1.0 micrometers at the next baking process is desirable. Moreover, although especially the ingredient that constitutes sealant 13a is not limited, either, the thing containing heat-resistant ingredients, such as an inorganic fiber and an inorganic binder, is desirable. Sealant 13a may be constituted by the same ingredient as a glue line 11.

0024 The ingredient which constitutes a glue line 11 contains an inorganic binder, an organic binder, and silicon carbide fiber. Although the bond strength of a glue line 11 is improved according to the effectiveness of a tangle to the silicon carbide fiber in a glue line 11, an inorganic binder, and silicon carbide fiber and an organic binder, both the bond strength of a glue line 11 and the heat conductivity are securable on high level by making silicon carbide fiber contain. It is thought by the reason being that the touch area of these silicon carbide fiber in the inside of a glue line increases since the silicon carbide to add is fibrous, and its heat conductivity improving compared with the silicon carbide particle added conventionally, and not checking effectiveness of a tangle to an inorganic binder etc. and becoming entangled with both reverse, since it is fibrous that the bond strength of a glue line 11 becomes high.

0025 As the above-mentioned inorganic binder, a silica sol, alumina sol, etc. are mentioned, for example. These may be used independently and may use two or more sorts together. In these, a silica sol is desirable.

0026 As the above-mentioned organic binder, polyvinyl alcohol, methyl cellulose, ethyl cellulose, a carboxy cellulose, etc. are mentioned, for example. These may be used independently and may use two or more sorts together. In these, a carboxy cellulose is desirable.

0027 As for the fiber length of the above-mentioned silicon carbide fiber, it is desirable that it is 20-300 micrometers, and it is more desirable that it is 50-200 micrometers. The property becomes it close to a particle that fiber length is less than 20 micrometers, and lowering of bond strength is caused. On the other hand, if it exceeds 300 micrometers, into a glue line, it will become difficult to distribute homogeneity and it will cause lowering of bond strength too. Moreover, as for the diameter of fiber, it is desirable that it is 3-15 micrometers. Since the reinforcement of silicon carbide fiber will fall that the diameter of fiber is less than 3 micrometers and it will be cut easily, lowering of bond strength is caused. On the other hand, if it exceeds 15 micrometers, it will be difficult to check a tangle to an inorganic binder, and to cause lowering of bond strength, and to

obtain such thick silicon carbide fiber itself, and it will cause the jump of raw material cost.
0028 It is solid content, the content of the inorganic binder in a glue line 11 has 1 - 40 desirable % of the weight, its 1 - 20 % of the weight is more desirable, and its 5 - 15 % of the weight is still more desirable. Lowering of bond strength is caused as the content of an inorganic binder is less than 1 % of the weight, and on the other hand, if it exceeds 40 % of the weight, decline in thermal conductivity will be caused.
0029 It is solid content, the content of the organic binder in a glue line 11 has 0.1 - 5.0 desirable % of the weight, its 0.2 - 1.0 % of the weight is more desirable, and its 0.4 - 0.8 % of the weight is still more desirable. It becomes difficult to control the migration of a glue line 11 as the content of an organic binder is less than 0.1 % of the weight, on the other hand, when it exceeded 5.0 % of the weight and a glue line 11 is exposed to an elevated temperature, an organic binder is burned down and bond strength falls.
0030 It is solid content, the content of the silicon carbide fiber in a glue line 11 has 3 - 80 desirable % of the weight, its 10 - 70 % of the weight is more desirable, and its 40 - 60 % of the weight is still more desirable. Decline in thermal conductivity is caused as the content of silicon carbide fiber is less than 3 % of the weight, and on the other hand, when it exceeded 80 % of the weight and a glue line 11 is exposed to an elevated temperature, lowering of bond strength is caused.
0031 Although little moisture, a little solvent, etc. may be included besides an inorganic binder, an organic binder, and silicon carbide fiber in the glue line 11, such moisture, a solvent, etc. usually almost disperse with heating after applying a glue line paste etc.
0032 Since it is what contains an inorganic binder, an organic binder, and silicon carbide fiber in the glue line to which the ceramic structure of this invention bands two or more ceramic members together as above-mentioned, it becomes the thing excellent in both the thermal conductivity and bond strength. Therefore, in the regeneration, the ceramic structure of this invention becomes what a crack arose in a glue line neither with an oscillation nor the pressure of exhaust gas, and was excellent in endurance while being able to carry out combustion clearance of the deposited particulate thoroughly.
0033 Next, the manufacture approach of the ceramic structure of this invention is explained. In addition, suppose that silicon carbide is used as a raw material of the ceramic member which constitutes the ceramic structure in the following explanation.
0034 A silicon carbide Plastic solid is produced first. After mixing silicon carbide powder, a binder, and dispersion-medium liquid and preparing the mixed constituent for Plastic solid manufacture in this process, by performing extrusion molding of this mixed constituent The column-like silicon carbide Plastic solid with which many breakthroughs separated the septum and were installed in the longitudinal direction side by side is produced, by drying this Plastic solid after this, dispersion-medium liquid is evaporated and the silicon carbide Plastic solid containing silicon carbide powder and resin is produced. In addition, little dispersion-medium liquid may be contained in this silicon carbide Plastic solid.
0035 The configuration of the appearance of this silicon carbide Plastic solid is isomorphism-like mostly with the porosity ceramic member 20 shown in drawing 2 , and also may have the shape of the shape of an elliptic cylinder, or the triangle pole etc. In addition, at this process, the part equivalent to a filler 22 serves as a cavity.
0036 It is not limited especially as the above-mentioned binder, for example, methyl cellulose, a carboxymethyl cellulose, hydroxyethyl cellulose, a polyethylene glycol, phenol resin, an epoxy resin, etc. can be mentioned. The loadings of the above-mentioned binder usually have desirable 1 - 10 weight section extent to the above-mentioned silicon carbide powder 100 weight section.
0037 It is not limited especially as the above-mentioned dispersion-medium liquid, for example, alcohol **, such as an organic solvent; methanol,** , such as benzene, water, etc. can be mentioned. Optimum dose combination of the above-mentioned dispersion-medium liquid is carried out so that the viscosity of the above-mentioned resin may become fixed within the limits.
0038 Next, the process which obturates the above-mentioned breakthrough of the produced silicon carbide Plastic solid in the shape of an obturation pattern with a restoration paste as an obturation process is performed. In this case, some breakthroughs are obturated with a restoration paste by contacting the breakthrough of a silicon carbide Plastic solid in the mask with which puncturing was formed in the shape of an obturation pattern, and making a restoration paste

invade into it from puncturing of the above-mentioned mask at the above-mentioned breakthrough.
0039 Or it will not be the mixed constituent and this appearance which were used as the above-mentioned restoration paste on the occasion of manufacture of a ceramic Plastic solid, what added the dispersion medium further to the above-mentioned mixed constituent is desirable.
0040 Next, the process which pyrolyzes the resin in the silicon carbide Plastic solid produced by the above-mentioned process as a cleaning process is performed. At this cleaning process, after laying the above-mentioned silicon carbide Plastic solid on the fixture for cleaning, it carries in to a cleaning furnace and usually heats at 400-650 degrees C under an oxygen content ambient atmosphere. Thereby, while resinous principles, such as a binder, vaporize, it decomposes and disappears and only silicon carbide powder remains mostly.
0041 Next, the process which lays the degreased silicon carbide Plastic solid on the fixture for baking, and calcinates it as a baking process is performed. At this baking process, the column-like silicon carbide sintered compact with which many breakthroughs separated the septum and were installed in the longitudinal direction side by side is manufactured by heating the silicon carbide Plastic solid degreased at 2000-2200 degrees C under inert gas ambient atmospheres, such as nitrogen and an argon, and making silicon carbide powder sinter.
0042 In addition, at a series of processes of resulting **from a cleaning process** in a baking process, the above-mentioned silicon carbide Plastic solid is carried on the fixture for baking, and it is desirable to perform a cleaning process and a baking process as it is. It is because it can prevent that can perform a cleaning process and a baking process efficiently, and carry, and a silicon carbide Plastic solid gets damaged in a substitute etc.
0043 Thus, many breakthroughs separate a septum and it is installed in a longitudinal direction side by side, and after manufacturing the porosity silicon carbide sintered compact constituted so that the above-mentioned septum might function as a filter, the glue line mentioned above into the outer wall part of a porosity silicon carbide sintered compact is formed as a union process of this porosity silicon carbide sintered compact, more than one band the above-mentioned porosity silicon carbide sintered compact together, and a hollow clay building block is produced so that it may become predetermined magnitude.
0044 Then, manufacture of the ceramic structure of this invention is ended by heating and stiffening **dry and** this hollow clay building block on 50-100 degrees C and the conditions of 1 hour, and forming sealant 13a in that periphery section, after cutting using after that, for example, a diamond cutter etc., almost like the ceramic structure 10 which showed that periphery section to drawing 1 .
0045 By carrying out each process explained above, thermal conductivity is high and the ceramic structure excellent also in the bond strength of each ceramic member can be manufactured.
0046
Example Although an example is hung up over below and this invention is explained to it in more detail, this invention is not limited only to these examples.
0047 The mixed constituent of a raw material was prepared by blending the alpha mold silicon carbide powder 70 weight section with example 1 mean particle diameter of 10 micrometers, the beta mold silicon carbide powder 30 weight section with a mean particle diameter of 0.7 micrometers, the methyl cellulose 5 weight section, the dispersant 4 weight section, and the water 20 weight section, and mixing to homogeneity. The extruding press machine was filled up with this mixed constituent, and the generation form of a honeycomb configuration was produced in a part for extrusion rate/of 2cm. This generation form is the same as that of the porosity ceramic member 20 shown in drawing 2 almost, that magnitude is 33mmx33mmx300mm, and, for an average pore diameter, the number of 1-40 micrometers and breakthroughs is 31-/cm2. The thickness of a septum was 0.35mm.
0048 After using the above-mentioned mixed constituent and the bulking agent paste of this component for the desiccation object of this generation form and filling it up with a bulking agent in the predetermined part of the breakthrough of a silicon carbide sintered compact, the porosity silicon carbide member was manufactured by degreasing at 450 degrees C and carrying out heating baking at 2200 degrees C further.
0049 Next, the carboxymethyl cellulose of 0.65 % of the weight, the fiber length of 100-200 micrometers, 44.2 % of the weight of silicon carbide fiber of 3-15 micrometers of diameters of

fiber, and 40.15 % of the weight of water were mixed and kneaded as an inorganic binder as 15 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, and an organic binder, and the paste for glue lines was prepared.

0050 Next, the above-mentioned paste for glue lines was stuck on the peripheral face of 1 of the produced porosity silicon carbide member, and the glue line was formed. And after laying other porosity silicon carbide members on this glue line, it was made to dry and harden in 100 degrees C and 1 hour, and the combination of the porosity silicon carbide member which two porosity silicon carbide members combined was produced.

0051 The carboxymethyl cellulose of 0.6 % of the weight, the fiber length of 100-200 micrometers, 60 % of the weight of silicon carbide fiber of 3-15 micrometers of diameters of fiber, and 30.4 % of the weight of water were used as an example 2 inorganic binder as 9 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, and an organic binder, and also the combination of a porosity silicon carbide member was produced like the example 1.

0052 The carboxymethyl cellulose of 0.6 % of the weight, the fiber length of 100-200 micrometers, 44.2 % of the weight of silicon carbide fiber of 3-15 micrometers of diameters of fiber, and 46.2 % of the weight of water were used as an example 3 inorganic binder as 9 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, and an organic binder, and also the combination of a porosity silicon carbide member was produced like the example 1.

0053 13.3 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols and 42.5 % of the weight of water were used as 44.2 % of the weight (shot content: 2.7%, fiber length:30-100mm) of ceramic fiber which consists of an alumina silica as example of comparison 1 inorganic fiber, and an inorganic binder, and also the combination of a porosity silicon carbide member was produced like the example 1. In this example 1 of a comparison, when drying the glue line, the migration of the binder which constitutes a glue line, or a member occurred, and the glue line became an ununiformity and has carried out.

0054 Ceramic fiber which consists of alumina silicate as example of comparison 2 inorganic fiber (shot content: 3%) As an inorganic binder, 23.3 0.1-100mm% of the weight Fiber length : 7 % of the weight (the content of SiO2 in a sol: 30 % of the weight) of silica sols, 0.5 % of the weight of carboxymethyl celluloses, 30.2 % of the weight of silicon carbide powder with a mean particle diameter of 0.3 micrometers, and 39 % of the weight of water were used as an organic binder, and also the combination of a porosity silicon carbide member was produced like the example 1.

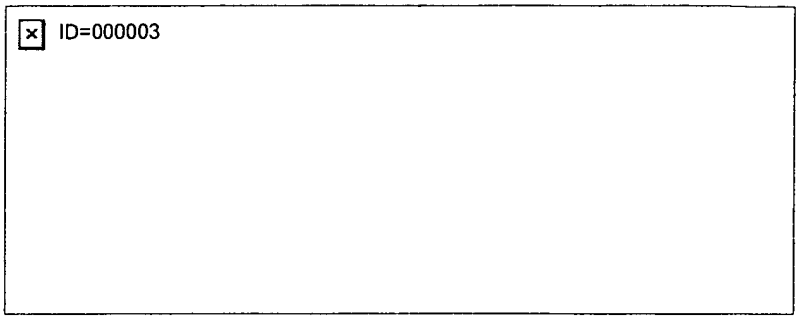
0055 It measured by the approach of showing below the performance evaluation of the combination of the porosity silicon carbide member manufactured in examples 1-3 and the examples 1-2 of a comparison.

0056 As shown in measurement drawing 3 of the assessment approach (1) bond strength, two triangle pole-like members have been arranged on a base, then the above-mentioned combination was laid so that the porosity silicon carbide member of ends might appear on the above-mentioned triangle pole-like member, the load was applied to the main glue line part, and the load when peeling arises in a glue line was measured. Moreover, in the actual activity, since the rapid heating to about room temperature -900 degree C and quenching were expected, assessment with the same said of the thing after performing the thermo-cycle trial (100 times) which is room temperature -900 degree C was performed. The result is shown in the following table 1.

0057 (2) After laying the above-mentioned combination so that two porosity silicon carbide members may be accumulated as shown in measurement drawing 4 of thermal conductivity, the temperature gradient of the upside temperature T1 and the lower temperature T2 was measured by installing the periphery on an enclosure and a heater 31 with a heat insulator 30, and heating for 30 minutes at 600 degrees C. The result is shown in a table 1.

0058

A table 1



0059 Although the typical bond strength of the glue line of the combination of the porosity silicon carbide member concerning examples 1-3 was 2.80-3.30MPa and the temperature gradient of the upper bed and soffit was 23-31 degrees C so that clearly from the result shown in a table 1, it was inferior to the combination of the porosity silicon carbide member which the typical bond strength of the glue line of the combination of the porosity silicon carbide member concerning the examples 1-2 of a comparison requires for 2.14-2.42MPa with 50-80 degrees C, and the temperature gradient all requires for an example. In addition, in this example and the example of a comparison, although what combined only two porosity silicon carbide members was used and the bond strength and thermal conductivity were measured, in order to combine many porosity silicon carbide members, the difference of the value of bond strength and thermal conductivity will become still more remarkable at the actual ceramic structure.

0060

Effect of the Invention It becomes what a crack arose in a glue line neither with an oscillation nor the pressure of exhaust gas, and was excellent in endurance while it can carry out combustion clearance of the deposited particulate thoroughly in the regeneration, since the ceramic structure of this invention is as above-mentioned.

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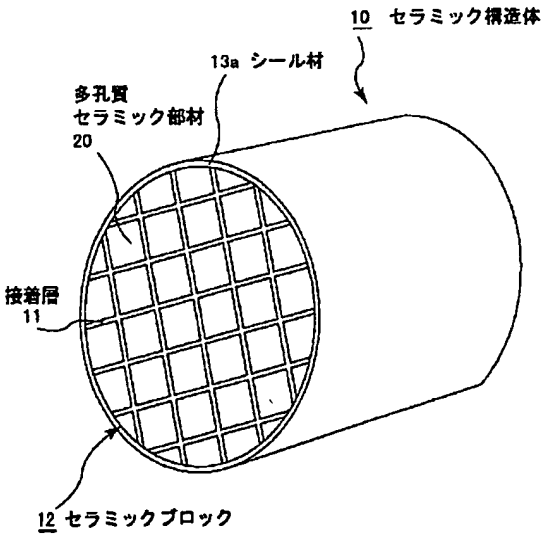
(71)出願人　000000158
イビデン株式会社
岐阜県大垣市神田町２丁目１番地
(72)発明者　大野　一茂
岐阜県揖斐郡揖斐川町北方１－１　イビデン株式会社大垣北工場内
(72)発明者　吉田　良行
岐阜県揖斐郡揖斐川町北方１－１　イビデン株式会社大垣北工場内
(74)代理人　100086586
弁理士　安富　康男　（外２名）

最終頁に続く

(54)【発明の名称】　セラミック構造体

(57)【要約】
【課題】　複数のセラミック部材を結束する接着層の熱伝導率が高いため、その再生処理において、堆積したパティキュレートを完全に燃焼除去することができるとともに、その接着強度も大きいため、振動や排気ガスの圧力等により接着層にクラック等が生ずることがない耐久性に優れるセラミック構造体を提供する。
【解決手段】　多数の貫通孔が隔壁を隔てて長手方向に並設された角柱形状の多孔質セラミック部材が接着層を介して複数個結束されてセラミックブロックを構成し、上記貫通孔を隔てる隔壁が粒子捕集用フィルタとして機能するように構成されたセラミック構造体であって、上記接着層は少なくとも無機バインダー、有機バインダー及び炭化珪素繊維を含むことを特徴とするセラミック構造体。



【特許請求の範囲】

【請求項１】　多数の貫通孔が隔壁を隔てて長手方向に並設された角柱形状の多孔質セラミック部材が接着層を介して複数個結束されてセラミックブロックを構成し、前記貫通孔を隔てる隔壁が粒子捕集用フィルタとして機能するように構成されたセラミック構造体であって、前記接着層は少なくとも無機バインダー、有機バインダー及び炭化珪素繊維を含むことを特徴とするセラミック構造体。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、内燃機関から排出される排気ガス中のパティキュレート等を除去するフィルタとして用いられるセラミック構造体に関する。

【０００２】

【従来の技術】バス、トラック等の車両や建設機械等の内燃機関から排出される排気ガス中に含有されるパティキュレートが環境や人体に害を及ぼすことが最近問題となっている。この排気ガスを多孔質セラミックを通過させることにより、排気ガス中のパティキュレートを捕集して排気ガスを浄化するセラミックフィルタが種々提案されている。

【０００３】これらのセラミックフィルタを構成するセラミック構造体は、通常、一方向に多数の貫通孔が並設され、貫通孔同士を隔てる隔壁がフィルタとして機能するようになっている。すなわち、セラミック構造体に形成された貫通孔は、排気ガスの入り口側又は出口側の端部のいずれかが充填材により目封じされ、一の貫通孔に流入した排気ガスは、必ず貫通孔を隔てる隔壁を通過した後、他の貫通孔から流出するようになっており、排気ガスがこの隔壁を通過する際、パティキュレートが隔壁部分で捕捉され、排気ガスが浄化される。

【０００４】このような排気ガスの浄化作用に伴い、セラミック構造体の貫通孔を隔てる隔壁部分には、次第にパティキュレートが堆積し、目詰まりを起こして通気を妨げるようになる。このため、このセラミックフィルタは、定期的にヒータ等の加熱手段を用いて目詰まりの原因となっているパティキュレートを燃焼除去して再生する必要がある。

【０００５】しかし、この再生処理においては、セラミック構造体の均一な加熱が難しく、パティキュレートの燃焼に伴う局部的な発熱が発生するため、大きな熱応力が発生する。また、通常の運転時においても、排気ガスの急激な温度変化が与える熱衝撃等によって、セラミック構造体の内部に不均一な温度分布が生じ、熱応力が発生する。その結果、上記セラミック構造体が単一のセラミック部材から構成されている場合には、クラックが発生し、パティキュレートの捕集に重大な支障を与えるといった問題点があった。

【０００６】そのため、例えば、特開昭６０－６５２１９号公報には、セラミック構造体を複数個のセラミック部材に分割することにより、セラミック構造体に作用する熱応力を低減させたパティキュレートトラップが開示されている。

【０００７】また、実開平１－６３７１５公報には、複数個のセラミック部材を結束させた際に、各部材の間に生じる隙間に、非接着性のシール材を介挿させ、セラミック構造体の隙間から排気ガスが漏れるのを防止した微粒子捕集フィルタが開示されている。

【０００８】しかし、この実開平１－６３７１５公報に開示された微粒子捕集フィルタでは、熱応力に起因するクラックの発生や破壊を防止することはできるが、各セラミック部材を強固に接合することができないという問題点があった。

【０００９】また、一般に、堆積したパティキュレートを燃焼除去する再生処理においては、セラミック構造体の中央付近の温度がその外縁部付近の温度に比べて高くなりやすい。しかしながら、従来のセラミック構造体では、該セラミック構造体を構成するセラミック部材間の熱伝導率が余り高くなかったため、その外縁部付近に堆積したパティキュレートが燃え残り、完全に除去することが困難であるという問題もあった。

【００１０】このような問題を解決するため、本発明者らは、先に、特開平８－２８２４６号公報に開示されているような、各セラミック部材が耐熱性の無機繊維や無機バインダー、有機バインダー及び無機粒子等を含むシール材（接着層）で接合されたセラミック構造体を開発した。

【００１１】このような、本発明者らが先に開発したセラミック構造体は、各セラミック部材同士を接合するシール材（接着層）中の無機繊維と有機バインダー、及び、無機繊維と無機バインダーとの絡み合いの効果により、ある程度接着強度が改善されるとともに、シール材（接着層）の熱伝導率も確保することができた。

【００１２】しかしながら、上記無機粒子は、無機繊維と無機バインダーとの絡み合いを阻害し、その接着強度を充分に高く保つことができないため、振動や排気ガスの圧力等により接着層にクラック等が生じてしまう場合があり、このようなセラミック構造体では、そのシール材（接着層）の接着強度と熱伝導率とを共に高いレベルで確保するには一定の限界があった。

【００１３】

【発明が解決しようとする課題】本発明は、これらの問題を解決するためになされたもので、複数のセラミック部材を結束する接着層の熱伝導率が高いため、その再生処理において、堆積したパティキュレートを完全に燃焼除去することができるとともに、その接着強度も大きいため、振動や排気ガスの圧力等により接着層にクラック等が生ずることのない耐久性に優れるセラミック構造体を提供することを目的とする。

【００１４】

【課題を解決するための手段】本発明のセラミック構造体は、多数の貫通孔が隔壁を隔てて長手方向に並設された角柱形状の多孔質セラミック部材が接着層を介して複数個結束されてセラミックブロックを構成し、上記貫通孔を隔てる隔壁が粒子捕集用フィルタとして機能するように構成されたセラミック構造体であって、上記接着層は少なくとも無機バインダー、有機バインダー及び炭化珪素繊維を含むことを特徴とするものである。

【００１５】

【発明の実施の形態】以下、本発明のセラミック構造体の実施形態について、図面に基づいて説明する。

【００１６】本発明のセラミック構造体は、多数の貫通孔が隔壁を隔てて長手方向に並設された角柱形状の多孔質セラミック部材が接着層を介して複数個結束されてセラミックブロックを構成し、上記貫通孔を隔てる隔壁が粒子捕集用フィルタとして機能するように構成されており、この接着層は少なくとも無機バインダー、有機バインダー及び炭化珪素繊維を含むものである。

【００１７】図１は、本発明のセラミック構造体の一実施形態を模式的に示した斜視図であり、図２は本発明のセラミック構造体を構成する多孔質セラミック部材を模式的に示した斜視図である。

【００１８】図２に示したように、セラミック構造体を構成する多孔質セラミック部材２０には、多数の貫通孔２１が形成されており、これら貫通孔２１を有する多孔質セラミック部材２０の一端部は、市松模様に充填材２２が充填されている。また、図示しない他の端部においては、一端部に充填材が充填されていない貫通孔２１に充填材が充填されている。

【００１９】図１は、図２に示した多孔質セラミック部材２０を複数個結束させたセラミック構造体１０を示している。また、図１においては、多孔質セラミック部材２０に形成された貫通孔２１を省略している。

【００２０】このセラミック構造体１０では、多孔質セラミック部材２０が接着層１１を介して複数個結束されてセラミックブロック１２を構成し、この接着層１１は、少なくとも無機バインダー、有機バインダー及び炭化珪素繊維を含むものである。また、セラミックブロック１２の外周部の全体に、シール材１３ａがコーティングされてセラミック構造体１０が形成されている。上記セラミック構造体の形状は特に限定されず、円柱形状でも角柱形状でも構わないが、通常、図１に示したように円柱形状のものがよく用いられている。

【００２１】このセラミック構造体１０を構成する多数の貫通孔２１は、図２に示したように、いずれか一端部のみに充填材２２が充填されているため、開口している一の貫通孔２１の一端部より流入した排気ガスは、隣接する貫通孔２１との間を隔てる多孔質の隔壁２３を必ず通過し、他の貫通孔２１を通って流出する。そして、排気ガスが隔壁２３を通過する際に、排気ガス中のパティキュレートが捕捉されることになる。

【００２２】上記セラミック構造体１０を構成する多孔質セラミック部材の材質は特に限定されず、種々のセラミックが挙げられるが、これらのなかでは、耐熱性が大きく、機械的特性に優れ、かつ、熱伝導率も大きい炭化珪素が好ましい。

【００２３】これらのセラミックの粒径も特に限定されるものではないが、後の焼成工程で収縮が少ないものが好ましく、例えば、０．３～５０μｍ程度の平均粒径を有する粉末１００重量部と０．１～１．０μｍ程度の平均粒径を有する粉末５～６５重量部とを組み合わせたものが好ましい。また、シール材１３ａを構成する材料も特に限定されるものではないが、無機繊維、無機バインダー等の耐熱性の材料を含むものが好ましい。シール材１３ａは、接着層１１と同じ材料により構成されていてもよい。

【００２４】接着層１１を構成する材料は、無機バインダー、有機バインダー及び炭化珪素繊維を含んでいる。接着層１１中の炭化珪素繊維と無機バインダー、及び、炭化珪素繊維と有機バインダーとの絡み合いの効果により、接着層１１の接着強度が改善されるが、炭化珪素繊維を含有させることにより、接着層１１の接着強度及び熱伝導率の両方を高いレベルで確保することができる。その理由は、添加する炭化珪素が繊維状であるため、接着層中でのこれら炭化珪素繊維同士の接触面積が増加することで、従来添加していた炭化珪素粒子に比べ熱伝導率が向上し、また、繊維状であるため、無機バインダー等との絡み合いの効果を阻害することがなく、逆に共に絡み合うことにより、接着層１１の接着強度が高くなるものと考えられる。

【００２５】上記無機バインダーとしては、例えば、シリカゾル、アルミナゾル等が挙げられる。これらは、単独で用いてもよく、２種以上を併用してもよい。これらのなかでは、シリカゾルが好ましい。

【００２６】上記有機バインダーとしては、例えば、ポリビニルアルコール、メチルセルロース、エチルセルロース、カルボキシセルロース等が挙げられる。これらは、単独で用いてもよく、２種以上を併用してもよい。これらのなかでは、カルボキシセルロースが好ましい。

【００２７】上記炭化珪素繊維の繊維長は、２０～３００μｍであることが好ましく、５０～２００μｍであることがより好ましい。繊維長が２０μｍ未満であると、その性質が粒子に近くなり接着強度の低下を招く。一方、３００μｍを超えると、接着層中に均一に分散させることが困難となり、やはり接着強度の低下を招く。また、その繊維径は、３～１５μｍであることが好ましい。繊維径が３μｍ未満であると、炭化珪素繊維の強度が低下し容易に切断されてしまうため接着強度の低下を招く。一方、１５μｍを超えると、無機バインダーとの

絡み合いを阻害し、接着強度の低下を招き、また、このような太い炭化珪素繊維を得ること自体が困難であり原料コストの高騰を招く。

【００２８】接着層１１中の無機バインダーの含有量は、固形分で、１～４０重量%が好ましく、１～２０重量%がより好ましく、５～１５重量%がさらに好ましい。無機バインダーの含有量が１重量%未満であると、接着強度の低下を招き、一方、４０重量%を超えると、熱伝導率の低下を招く。

【００２９】接着層１１中の有機バインダーの含有量は、固形分で、０．１～５．０重量%が好ましく、０．２～１．０重量%がより好ましく、０．４～０．８重量%がさらに好ましい。有機バインダーの含有量が０．１重量%未満であると、接着層１１のマイグレーションを抑制するのが難しくなり、一方、５．０重量%を超えると、接着層１１が高温にさらされた場合に、有機バインダーが焼失し、接着強度が低下する。

【００３０】接着層１１中の炭化珪素繊維の含有量は、固形分で、３～８０重量%が好ましく、１０～７０重量%がより好ましく、４０～６０重量%がさらに好ましい。炭化珪素繊維の含有量が３重量%未満であると、熱伝導率の低下を招き、一方、８０重量%を超えると、接着層１１が高温にさらされた場合に、接着強度の低下を招く。

【００３１】接着層１１中には、無機バインダー、有機バインダー及び炭化珪素繊維のほかに、少量の水分や溶剤等を含んでいてもよいが、このような水分や溶剤等は、通常、接着層ペーストを塗布した後の加熱等により殆ど飛散する。

【００３２】上述の通り、本発明のセラミック構造体は、複数のセラミック部材を結束する接着層中に、無機バインダー、有機バインダー及び炭化珪素繊維を含むものであるため、その熱伝導率及び接着強度の両方に優れたものとなる。従って、本発明のセラミック構造体は、その再生処理において、堆積したパティキュレートを完全に燃焼除去することができるとともに、振動や排気ガスの圧力等により接着層にクラックが生ずることはなく、耐久性に優れたものとなる。

【００３３】次に、本発明のセラミック構造体の製造方法について説明する。なお、以下の説明においては、セラミック構造体を構成するセラミック部材の原料として炭化珪素を用いることとする。

【００３４】初めに、まず、炭化珪素成形体を作製する。この工程においては、炭化珪素粉末とバインダーと分散媒液とを混合して成形体製造用の混合組成物を調製した後、この混合組成物の押出成形を行うことにより、多数の貫通孔が隔壁を隔てて長手方向に並設された柱状の炭化珪素成形体を作製し、この後、この成形体を乾燥させることにより分散媒液を蒸発させ、炭化珪素粉末と樹脂とを含む炭化珪素成形体を作製する。なお、この炭化珪素成形体には、少量の分散媒液が含まれていてもよい。

【００３５】この炭化珪素成形体の外観の形状は、図２に示した多孔質セラミック部材２０とほぼ同形状であるほか、楕円柱状や三角柱状等であってもよい。なお、本工程では、充填材２２に相当する部分は空洞となっている。

【００３６】上記バインダーとしては特に限定されず、例えば、メチルセルロース、カルボキシメチルセルロース、ヒドロキシエチルセルロース、ポリエチレングリコール、フェノール樹脂、エポキシ樹脂等を挙げることができる。上記バインダーの配合量は、通常、上記炭化珪素粉末１００重量部に対して、１～１０重量部程度が好ましい。

【００３７】上記分散媒液としては特に限定されず、例えば、ベンゼン等の有機溶媒；メタノール等のアルコール、水等を挙げることができる。上記分散媒液は、上記樹脂の粘度が一定範囲内となるように、適量配合される。

【００３８】次に、封口工程として、作製された炭化珪素成形体の上記貫通孔を充填ペーストにより封口パターン状に封口する工程を行う。この際には、炭化珪素成形体の貫通孔に、封口パターン状に開孔が形成されたマスクを当接し、充填ペーストを上記マスクの開孔から上記貫通孔に侵入させることにより、充填ペーストで一部の貫通孔を封口する。

【００３９】上記充填ペーストとしては、セラミック成形体の製造の際に使用した混合組成物と同様のものか、又は、上記混合組成物にさらに分散媒を添加したものが好ましい。

【００４０】次に、脱脂工程として、上記工程により作製された炭化珪素成形体中の樹脂を熱分解する工程を行う。この脱脂工程では、通常、上記炭化珪素成形体を脱脂用治具上に載置した後、脱脂炉に搬入し、酸素含有雰囲気下、４００～６５０℃に加熱する。これにより、バインダー等の樹脂成分が揮散するとともに、分解、消失し、ほぼ炭化珪素粉末のみが残留する。

【００４１】次に、焼成工程として、脱脂した炭化珪素成形体を、焼成用治具上に載置して焼成する工程を行う。この焼成工程では、窒素、アルゴン等の不活性ガス雰囲気下、２０００～２２００℃で脱脂した炭化珪素成形体を加熱し、炭化珪素粉末を焼結させることにより、多数の貫通孔が隔壁を隔てて長手方向に並設された柱状の炭化珪素焼結体を製造する。

【００４２】なお、脱脂工程から焼成工程に至る一連の工程では、焼成用治具上に上記炭化珪素成形体を載せ、そのまま、脱脂工程及び焼成工程を行うことが好ましい。脱脂工程及び焼成工程を効率的に行うことができ、また、載せ代え等において、炭化珪素成形体が傷つくのを防止することができるからである。

【００４３】このようにして、多数の貫通孔が隔壁を隔てて長手方向に並設され、上記隔壁がフィルタとして機能するように構成された多孔質炭化珪素焼結体を製造した後、この多孔質炭化珪素焼結体の結束工程として、多孔質炭化珪素焼結体の外壁部分に上述した接着層を形成し、所定の大きさになるように上記多孔質炭化珪素焼結体を複数個結束してセラミックブロックを作製する。

【００４４】その後、このセラミックブロックを５０～１００℃、１時間の条件で加熱して乾燥、硬化させ、その後、例えば、ダイヤモンドカッター等を用いて、その外周部を図１に示したセラミック構造体１０とほぼ同様に切削した後、その外周部にシール材１３ａを形成することにより、本発明のセラミック構造体の製造を終了する。

【００４５】以上説明した各工程を実施することで、熱伝導率が高く、各セラミック部材の接着強度にも優れたセラミック構造体を製造することができる。

【００４６】

【実施例】以下に実施例を掲げて本発明を更に詳しく説明するが、本発明はこれら実施例のみに限定されるものではない。

【００４７】実施例１

平均粒径１０μｍのα型炭化珪素粉末７０重量部、平均粒径０．７μｍのβ型炭化珪素粉末３０重量部、メチルセルロース５重量部、分散剤４重量部、水２０重量部を配合して均一に混合することにより、原料の混合組成物を調製した。この混合組成物を押出成形機に充填し、押出速度２ｃｍ／分にてハニカム形状の生成形体を作製した。この生成形体は、図２に示した多孔質セラミック部材２０とほぼ同様であり、その大きさは３３ｍｍ×３３ｍｍ×３００ｍｍで、平均気孔径が１～４０μｍ、貫通孔の数が３１／ｃｍ^{2}で、隔壁の厚さが０．３５ｍｍであった。

【００４８】この生成形体の乾燥体に、上記混合組成物と同成分の充填剤ペーストを用いて、炭化珪素焼結体の貫通孔の所定箇所に充填剤を充填した後、４５０℃で脱脂し、さらに、２２００℃で加熱焼成することで多孔質炭化珪素部材を製造した。

【００４９】次に、無機バインダーとしてシリカゾル（ゾル中のSiO_2の含有量：３０重量%）１５重量%、有機バインダーとしてカルボキシメチルセルロース０．６５重量%、繊維長１００～２００μｍ、繊維径３～１５μｍの炭化珪素繊維４４．２重量%、及び、水４０．１５重量%を混合、混練して接着層用ペーストを調製した。

【００５０】次に、作製した多孔質炭化珪素部材の一の外周面に上記接着層用ペーストを貼着し、接着層を形成した。そして、この接着層の上に他の多孔質炭化珪素部材を載置した後、１００℃、１時間で乾燥、硬化させ、２つの多孔質炭化珪素部材が結合した多孔質炭化珪素部材の結合体を作製した。

【００５１】実施例２

無機バインダーとしてシリカゾル（ゾル中のSiO_2の含有量：３０重量%）９重量%、有機バインダーとしてカルボキシメチルセルロース０．６重量%、繊維長１００～２００μｍ、繊維径３～１５μｍの炭化珪素繊維６０重量%、及び、水３０．４重量%を用いたほかは、実施例１と同様にして多孔質炭化珪素部材の結合体を作製した。

【００５２】実施例３

無機バインダーとしてシリカゾル（ゾル中のSiO_2の含有量：３０重量%）９重量%、有機バインダーとしてカルボキシメチルセルロース０．６重量%、繊維長１００～２００μｍ、繊維径３～１５μｍの炭化珪素繊維４４．２重量%、及び、水４６．２重量%を用いたほかは、実施例１と同様にして多孔質炭化珪素部材の結合体を作製した。

【００５３】比較例１

無機繊維としてアルミナシリカからなるセラミックファイバー（ショット含有率：２．７%、繊維長：３０～１００ｍｍ）４４．２重量%、無機バインダーとしてシリカゾル（ゾル中のSiO_2の含有量：３０重量%）１３．３重量%および水４２．５重量%を用いたほかは、実施例１と同様にして多孔質炭化珪素部材の結合体を作製した。本比較例１においては、接着層の乾燥を行っている際、接着層を構成するバインダーや部材のマイグレーションが発生し、接着層が不均一になってしてしまった。

【００５４】比較例２

無機繊維としてアルミナシリケートからなるセラミックファイバー（ショット含有率：３%、繊維長：０．１～１００ｍｍ）２３．３重量%、無機バインダーとしてシリカゾル（ゾル中のSiO_2の含有量：３０重量%）７重量%、有機バインダーとしてカルボキシメチルセルロース０．５重量%、平均粒径０．３μｍの炭化珪素粉末３０．２重量%、及び、水３９重量%を用いたほかは、実施例１と同様にして多孔質炭化珪素部材の結合体を作製した。

【００５５】実施例１～３及び比較例１～２で製造した多孔質炭化珪素部材の結合体の性能評価を以下に示す方法にて測定した。

【００５６】<u>評価方法</u>

（１）接着強度の測定

図３に示すように、台の上に２個の三角柱状部材を配置し、続いて、上記結合体を、両端の多孔質炭化珪素部材が上記三角柱状部材の上に載るように載置し、中心の接着層部分に荷重をかけ、接着層に剥がれが生じた時の荷重を測定した。また、実際の使用では、室温～９００℃程度までの急熱、急冷が予想されるため、室温～９００℃のヒートサイクル試験（１００回）を行った後のもの

についても同様の評価を行った。その結果を下記の表１に示す。

【００５７】（２）熱伝導率の測定

図４に示すように、上記結合体を、２個の多孔質炭化珪素部材を積み重ねるように載置した後、その外周を断熱材３０で囲い、ヒータ３１の上に設置して６００℃で３０分間加熱することにより、上部の温度Ｔ１と下部の温度Ｔ２との温度差を測定した。その結果を表１に示す。

【００５８】

【表１】

	初期状態の接着強度（ＭＰａ）	ﾋｰﾄｻｲｸﾙ 後の接着強度（ＭＰａ）	T1-T2 温度差（℃）
実施例１	３．３０	３．１５	３１
実施例２	２．８０	２．６９	２３
実施例３	３．１５	３．０５	２６
比較例１	２．４２	２．３５	８０
比較例２	２．１４	２．０５	５０

【００５９】表１に示した結果から明らかなように、実施例１～３に係る多孔質炭化珪素部材の結合体の接着層の代表的な接着強度は２．８０～３．３０ＭＰａであり、その上端と下端との温度差は２３～３１℃であるが、比較例１～２に係る多孔質炭化珪素部材の結合体の接着層の代表的な接着強度は２．１４～２．４２ＭＰａ、その温度差は５０～８０℃といずれも、実施例に係る多孔質炭化珪素部材の結合体よりも劣ったものであった。なお、本実施例及び比較例においては、多孔質炭化珪素部材を２個だけ結合したものを使用して、その接着強度及び熱伝導率を測定したが、実際のセラミック構造体には、多数の多孔質炭化珪素部材を結合するため、接着強度及び熱伝導率の値の差はさらに顕著なものとなる。

【００６０】

【発明の効果】本発明のセラミック構造体は、上述の通りであるので、その再生処理において、堆積したパティキュレートを完全に燃焼除去することができるとともに、振動や排気ガスの圧力等によって接着層にクラックが生ずることがなく耐久性に優れたものとなる。

【図面の簡単な説明】

【図１】本発明のセラミック構造体の一実施形態を模式的に示した斜視図である。

【図２】本発明のセラミック構造体を構成する多孔質セラミック部材を模式的に示した斜視図である。

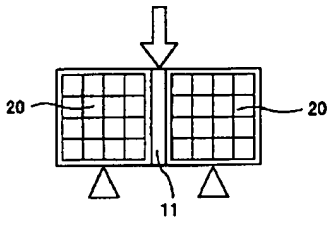
【図３】接着強度の測定試験の説明図である。

【図４】熱伝導率の測定試験の説明図である。

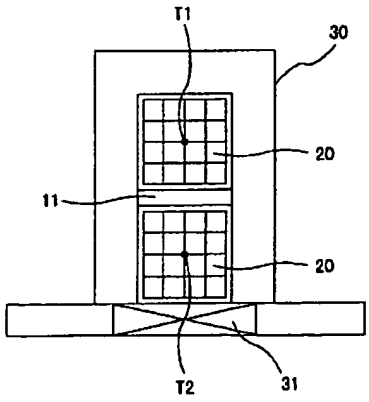
【符号の説明】

１０　セラミック構造体
１１　接着層
１２　セラミックブロック
１３　シール材ペースト
１３ａ　シール材
２０　多孔質セラミック部材
２１　貫通孔
２２　充填材
２３　隔壁
３０　断熱材
３１　ヒータ

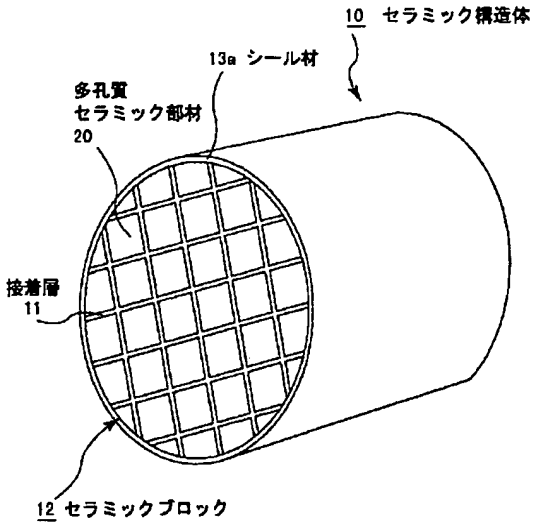
【図３】



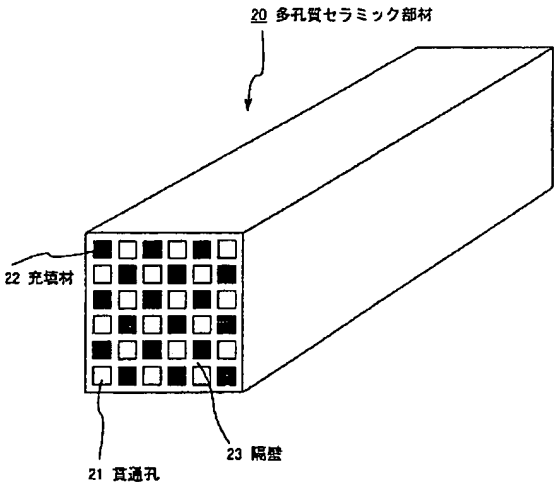
【図４】



【図１】



【図２】



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